

East Building, PHH-30 1200 New Jersey Avenue S.E. Washington, D.C. 20590

Pipeline and Hazardous Materials Safety Administration

DOT-SP 13220 (FIFTH REVISION)

EXPIRATION DATE: September 30, 2018

(FOR RENEWAL, SEE 49 CFR § 107.109)

1. <u>GRANTEE</u>: Entegris Inc. Danbury, CT

2. PURPOSE AND LIMITATION:

- a. This special permit authorizes the transportation in commerce of certain non-DOT specification welded pressure vessels containing certain compressed gases and liquids adsorbed onto a microporous substrate. This special permit provides no relief from the Hazardous Materials Regulations (HMR) or the International Maritime Dangerous Goods (IMDG) Code, as applicable, for the safe transport of dangerous goods other than as specifically stated herein. The most recent revision supersedes all previous revisions.
- b. The safety analyses performed in development of this special permit only considered the hazards and risks associated with transportation in commerce.
- c. Party status will not be granted under this special permit.
- d. This special permit serves as an "Approval" under Chapter 4.1, Section 4.1.3.7 and Chapter 7.9, Section 7.9.2 of the IMDG Code, and as a "Competent Authority Approval" as defined in 49 CFR 107.1.
- 3. <u>REGULATORY SYSTEM AFFECTED</u>: 49 CFR Parts 106, 107 and 171-180 and the IMDG Code.

- 4. REGULATIONS FROM WHICH EXEMPTED: 49 CFR §§ 173.192, 173.302a and 173.304a in that a non-DOT specification cylinder is not authorized, except as specified herein, and Packing Instruction P200 and P208 of the IMDG Code in that a non-UN standard cylinder is not authorized, except as stated herein.
- 5. <u>BASIS</u>: This special permit is based on the application of Entegris, Inc. dated October 6, 2014 and additional information dated April 1, 2015 submitted in accordance with § 107.105.

6. HAZARDOUS MATERIALS (49 CFR § 172.101):

| Hazardous Materials Description | | | | |
|--|------------------------------|-------------------------------|-------------------------------|--|
| Proper Shipping Name | Hazard Class/ Division | Identi- fication Number | Packing Group | |
| Adsorbed gas, n.o.s | 2.2 | UN3512 | N/A | |
| Adsorbed gas, flammable, n.o.s | 2.1 | UN3510 | N/A | |
| Adsorbed gas, oxidizing, n.o.s | 2.2 | UN3513 | N/A | |
| Adsorbed gas, toxic, n.o.s | 2.3 | UN3511 | N/A Hazard Zone A,B,C&D | |
| Adsorbed gas, toxic, corrosive, n.o.s | 2.3 | UN3516 | N/A Hazard Zone A,B,C&D | |
| Adsorbed gas, toxic, flammable, n.o.s | 2.3 | UN3514 | N/A Hazard Zone A,B,C&D | |
| Adsorbed gas, toxic, flammable, corrosive, n.o.s | 2.3 | UN3517 | N/A Hazard Zone A,B,C&D | |

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| Hazardous Materials Description | | | | |
|--|------------------------------|-------------------------------|-------------------------------|--|
| Proper Shipping Name | Hazard Class/ Division | Identi- fication Number | Packing Group | |
| Adsorbed gas, toxic, oxidizing, n.o.s | 2.3 | UN3515 | N/A Hazard Zone A,B,C&D | |
| Adsorbed gas, toxic, oxidizing, corrosive, n.o.s | 2.3 | UN3518 | N/A Hazard Zone A,B,C&D | |
| Ammonia Anhydrous | 2.3 | UN1005 | N/A Hazard Zone D | |
| Arsine | 2.3 | UN2188 | N/A Hazard Zone A | |
| Arsine, adsorbed | 2.3 | UN3522 | N/A Hazard Zone A | |
| Boron trichloride | 2.3 | UN1741 | N/A Hazard Zone C | |
| Boron trifluoride, adsorbed | 2.3 | UN3519 | N/A Hazard Zone B | |
| Boron trifluoride, compressed | 2.3 | UN1008 | N/A Hazard Zone B | |
| Carbon monoxide, compressed | 2.3 | UN1016 | N/A Hazard Zone D | |
| Chlorine | 2.3 | UN1017 | N/A Hazard Zone B | |
| Chlorine, adsorbed | 2.3 | UN3520 | N/A Hazard Zone B | |
| Compressed gas, n.o.s. | 2.2 | UN1956 | N/A | |
| Compressed gas, flammable, n.o.s. | 2.1 | UN1954 | N/A | |

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| Hazardous Materials Description | | | | |
|--|------------------------------|-------------------------------|----------------------|--|
| Proper Shipping Name | Hazard Class/ Division | Identi- fication Number | Packing Group | |
| Compressed gas, toxic, n.o.s. | 2.3 | UN1955 | N/A Hazard Zone A | |
| Compressed gas, toxic, corrosive, n.o.s. | 2.3 | UN3304 | N/A Hazard Zone B | |
| Dichlorosilane | 2.3 | UN2189 | N/A Hazard Zone B | |
| Germane | 2.3 | UN2192 | N/A Hazard Zone B | |
| Germane, absorbed | 2.3 | UN3523 | N/A Hazard Zone A | |
| Hydrogen bromide anhydrous | 2.3 | UN1048 | N/A Hazard Zone C | |
| Hydrogen chloride anhydrous | 2.3 | UN1050 | N/A Hazard Zone B | |
| Hydrogen selenide, adsorbed | 2.3 | UN3526 | N/A Hazard Zone A | |
| Hydrogen selenide, anhydrous | 2.3 | UN2202 | N/A Hazard Zone A | |
| Hydrogen sulfide | 2.3 | UN1053 | N/A Hazard | |
| Methyl mercaptan | 2.3 | UN1064 | N/A Hazard Zone C | |
| Nitric oxide, compressed | 2.3 | UN1660 | N/A Hazard Zone A | |
| Phosgene | 2.3 | UN1076 | N/A Hazard Zone A | |
| Phosphorus pentafluoride, compressed | 2.3 | UN2198 | N/A Hazard Zone B | |

| Hazardous Materials Description | | | | |
|---|------------------------------|-------------------------------|-----------------------|--|
| Proper Shipping Name | Hazard Class/ Division | Identi- fication Number | Packing Group | |
| Phosphorous pentafluoride, adsorbed | 2.3 | UN3524 | N/A Hazard Zone B | |
| Phosphine | 2.3 | UN2199 | N/A Hazard Zone A | |
| Phosphine, adsorbed | 2.3 | UN3525 | N/A Hazard Zone A | |
| Silane, compressed | 2.1 | UN2203 | N/A | |
| Silicon tetrafluoride, adsorbed | 2.3 | UN3521 | N/A Hazard Zone B | |
| Silicon tetrafluoride, compressed | 2.3 | UN1859 | N/A Hazard Zone B | |
| Sulfur dioxide | 2.3 | UN1079 | N/A Hazard Zone C | |
| Toxic liquids, flammable, organic, n.o.s. | 6.1 | UN2929 | PG I Hazard Zone B | |
| Tungsten hexafluoride | 2.3 | UN2196 | N/A Hazard Zone B | |

^{*}Note: New UN classification for these gases in adsorbed conditions have been added to this table. The shipment of these adsorbed gases under their new UN adsorbed gas designation shall be mandatory from January 1, 2016.

7. SAFETY CONTROL MEASURES:

a. PACKAGING - Packaging prescribed is a non-DOT specification, welded pressure vessel filled with a monolith solid microporous sorbent and/or bead type sorbent onto which the gas is adsorbed. The gas remains adsorbed during transportation in essentially a solid state. The system is filled and operated at sub-atmospheric pressures and is described as a sub-atmospheric gas delivery system (SDS). The welded pressure vessel may be of cylindrical or cube

design. The pressure vessel must be manufactured from cold drawn over mandrel (square shell) and/or cold deep drawn (cylindrical shell) steel. The pressure vessel must be designed and constructed in accordance with American Cap Company, Inc. drawing AC2314-C Rev J, AC2322-E Rev A, or AC2389 on file with the Office of Hazardous Materials Special Permits and Approvals (OHMSPA) and with the following specifications:

Capacity: 0.4 L to 12 Liters

Material: Steel Shell: ASTM Grade

A-1011/A-1011M-04A, DS Type A modified, hot rolled, steel with max carbon content 0.25%; or cold deep drawn AISI 304/304L Stainless steel

sheet

End Plug: Hot Rolled Steel SAE 1020 modified with max. carbon content

0.25%; or AISI 316L stainless steel bar

Minimum sidewall thickness: Cylindrical, 0.085 inch

Cube, 0.12 inch

Maximum service pressure: 75 psig

Minimum test pressure: 300 psig

Minimum design burst pressure: 1371 psig

(1) The pressure vessel must be manufactured in accordance with § 178.35 except as follows:

§ 178.35(b) (2) Inspections and verifications must be performed by a competent inspector of the manufacturer.

§ 178.35(f)(1)(i) The pressure vessel must be marked "DOT-SP 13220" followed by the service pressure in lieu of marking the DOT specification number. Markings must be stamped plainly and permanently in accordance with § 178.51(n).

- (2) The pressure vessel must be equipped with pressure relief devices as prescribed in § 173.301(f) for the hazardous material being transported except that compliance with Compressed Gas Association Pamphlet S-1.1 referenced in § 171.7 (compliance with paragraph 9.1.1.1 is not required). The selection of the type, location and quantity of a pressure relief device for an adsorbed gas package is based on the name of the gas in the unabsorbed state. For example the selection of a pressure relief device for "UN 3519 Boron trifluoride, adsorbed" would be based on the pressure relief device required for Baron Trifluoride, UN 1008. Valve protection must be in accordance with § 173.301(h).
- (3) Pressure vessels must be manufactured in accordance with American Cap Company Inc., Cylinder Manufacturing Procedure Doc. No. 10-007 and SDS Manufacturing Workflow Chart.
- (4) Welding or brazing must be by automated processes. Welding procedures and operators must be qualified in accordance with CGA Pamphlet C-3. Welded pressure vessels containing the sorbent material are not heat treated after the welding or brazing operation.
- (5) Test of welds must be performed in accordance with § 178.51(1).
- (6) At least one completed pressure vessel selected at random per lot of 200 or less must be hydrostatically tested to failure or to at least 1371 psig. The test will be performed without sorbent present. The pressure vessel must not show evidence of leakage or damage below 1371 psig. All pressure vessels used in the burst test must be destroyed.
- b. <u>REQUALIFICATION</u> Each pressure vessel must be requalified, every 10 years as follows:
 - (1) The pressure vessel must be evacuated and filled with Helium, to at least 300 psig. The pressure vessel must be placed in a vacuum chamber and the chamber evacuated to a pressure sufficient for the leak detector to meet the leak test specification. The pressure vessel must be held in the chamber until the leak detector provides a "ready to test" signal and the leak rate determined. The leak detection equipment must be calibrated for each test with nominal detection

limits of 1 x 10^{-10} atm. cc/sec. The pressure vessel must be rejected if the leak rate is greater than 1x 10^{-8} atm. cc/sec.

(2) The requalifer must comply with the marking and record keeping requirements of §§ 180.213 and 180.215.

c. OPERATIONAL CONTROLS -

- (1) Prior to the first filling, each completed pressure vessel with sorbent must be vacuum baked at a maximum temperature of 180°C to remove any contaminants.
- (2) Prior to filling with the gas, each completed pressure vessel containing the sorbent and with the valve attached must be proof pressure tested to at least 300 psig and helium leak tested per the test method in Paragraph 7.b. of this special permit.
- (3) Only Entegris Inc. or its agents authorized and trained by Entegris Inc. may fill the pressure vessels.
- (4) The maximum pressure inside the pressure vessel at $140^{\circ}F$ must not exceed 30 psig.
- (5) Each pressure vessel must remain in dedicated product service for its entire life.

8. SPECIAL PROVISIONS:

- a. A person who is not a holder of this special permit who receives a package covered by this special permit may reoffer it for transportation provided no modification or change is made to the package and it is reoffered for transportation in conformance with this special permit and the HMR.
- b. A current copy of this special permit must be maintained at each facility where the package is offered or reoffered for transportation. Offerers of empty cylinders (containing no residual hazardous material) being returned for refilling are not required to have copies of this special permit.
- 9. MODES OF TRANSPORTATION AUTHORIZED: Motor vehicle, rail freight and cargo vessel.

- 10. MODAL REQUIREMENTS: A current copy of this special permit must be carried aboard each cargo vessel or motor vehicle used to transport packages covered by this special permit.
- 11. <u>COMPLIANCE</u>: Failure by a person to comply with any of the following may result in suspension or revocation of this special permit and penalties prescribed by the Federal hazardous materials transportation law, 49 U.S.C. 5101 <u>et seq</u>:
 - o All terms and conditions prescribed in this special permit and the Hazardous Materials Regulations, 49 CFR Parts 171-180.
 - o Persons operating under the terms of this special permit must comply with the security plan requirement in Subpart I of Part 172 of the HMR, when applicable.
 - o Registration required by § 107.601 et seq., when applicable.

Each "Hazmat employee", as defined in § 171.8, who performs a function subject to this special permit must receive training on the requirements and conditions of this special permit in addition to the training required by §§ 172.700 through 172.704.

No person may use or apply this special permit, including display of its number, when this special permit has expired or is otherwise no longer in effect.

Under Title VII of the Safe, Accountable, Flexible, Efficient Transportation Equity Act: A Legacy for Users (SAFETEA-LU) - "The Hazardous Materials Safety and Security Reauthorization Act of 2005" (Pub. L. 109-59), 119 Stat. 1144 (August 10, 2005), amended the Federal hazardous materials transportation law by changing the term "exemption" to "special permit" and authorizes a special permit to be granted up to two years for new special permits and up to four years for renewals.

12. REPORTING REQUIREMENTS: Shipments or operations conducted under this special permit are subject to the Hazardous Materials Incident Reporting requirements specified in 49 CFR §§ 171.15 Immediate notice of certain hazardous materials incidents, and 171.16 Detailed hazardous materials incident reports. In addition, the grantee(s) of this special permit must notify the Associate Administrator for Hazardous Materials Safety, in writing, of any incident involving a package, shipment or operation conducted under terms of this special permit.

Issued in Washington, D.C.:

Dona By

for Dr. Magdy El-Sibaie

Associate Administrator for Hazardous Materials Safety

Address all inquiries to: Associate Administrator for Hazardous Materials Safety, Pipeline and Hazardous Materials Safety Administration, Department of Transportation, Washington, D.C. 20590. Attention: PHH-31.

Copies of this special permit may be obtained by accessing the Hazardous Materials Safety Homepage at http://hazmat.dot.gov/sp_app/special_permits/spec_perm_index.htm
Photo reproductions and legible reductions of this special permit are permitted. Any alteration of this special permit is prohibited.

PO: CWFreeman/RS/SG